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UTILITY PATENT APPLICATION TRANSMITTAL

(Only for new nonprovisional applications under 37 C.F.R. § 1.53(b))

Attorney Docket No. IB-1366
 First Inventor or Application Identifier Charles Piskoti et al.
 Title Carbon Based Thirty Six Atom Spheres
 Express Mail Label No. EL252172355US

APPLICATION ELEMENTS

See MPEP chapter 600 concerning utility patent application contents.

1. ☐ * Fee Transmittal Form (e.g., PTO/SB/17)
(Submit an original and a duplicate for fee processing)
2. ☒ Specification [Total Pages 15]
(preferred arrangement set forth below)
 - Descriptive title of the Invention
 - Cross References to Related Applications
 - Statement Regarding Fed sponsored R & D
 - Reference to Microfiche Appendix
 - Background of the Invention
 - Brief Summary of the Invention
 - Brief Description of the Drawings (if filed)
 - Detailed Description
 - Claim(s)
 - Abstract of the Disclosure
3. ☒ Drawing(s) (35 U.S.C. 113) [Total Sheets 9]
4. Oath or Declaration [Total Pages 1]
 - a. ☐ Newly executed (original or copy)
 - b. ☐ Copy from a prior application (37 C.F.R. § 1.63(d))
(for continuation/divisional with Box 16 completed)
 - i. ☐ DELETION OF INVENTOR(S)
Signed statement attached deleting inventor(s) named in the prior application, see 37 C.F.R. §§ 1.63(d)(2) and 1.33(b).

*NOTE FOR ITEMS 1 & 13: IN ORDER TO BE ENTITLED TO PAY SMALL ENTITY FEES, A SMALL ENTITY STATEMENT IS REQUIRED (37 C.F.R. § 1.27), EXCEPT IF ONE FILED IN A PRIOR APPLICATION IS RELIED UPON (37 C.F.R. § 1.28).

ADDRESS TO: Assistant Commissioner for Patents
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5. ☐ Microfiche Computer Program (Appendix)
6. Nucleotide and/or Amino Acid Sequence Submission (if applicable, all necessary)
 - a. ☐ Computer Readable Copy
 - b. ☐ Paper Copy (identical to computer copy)
 - c. ☐ Statement verifying identity of above copies

ACCOMPANYING APPLICATION PARTS

7. ☐ Assignment Papers (cover sheet & document(s))
8. ☐ 37 C.F.R. § 3.73(b) Statement of Power of Attorney (when there is an assignee)
9. ☐ English Translation Document (if applicable)
10. ☐ Information Disclosure Statement (IDS)/PTO-1449 [Copies of IDS Citations]
11. ☐ Preliminary Amendment
12. ☒ Return Receipt Postcard (MPEP 503)
(Should be specifically itemized)
13. ☐ * Small Entity Statement(s) [Statement filed in prior application, Status still proper and desired (PTO/SB/09-12)]
14. ☐ Certified Copy of Priority Document(s) (if foreign priority is claimed)
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16. If a CONTINUING APPLICATION, check appropriate box, and supply the requisite information below and in a preliminary amendment:

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Prior application information: Examiner _____ Group / Art Unit: _____

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IB-1366

Carbon Based Thirty Six Atom Spheres

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Carbon Based Thirty Six Atom Spheres

Related Applications

This application claims priority of Provisional Application Ser. No. 60/123,112 filed 03/05/99 which is herein incorporated by reference.

Government Rights

The United States Government has rights in this invention pursuant to Contract No.DE-AC03-76SF00098 between the United States Department of Energy and the University of California.

Background of the Invention

The invention relates to carbon spheres or fullerenes based on thirty six carbon atoms, and more particularly to solid state materials formed of these fullerenes and methods of making same.

The discovery in 1985 of a new form of carbon, the pure carbon cage molecule C₆₀, known as the "Bucky Ball" or [Buckminster]fullerene, initiated a new class of materials. In its molecular form C₆₀ is a highly spherical hollow cage molecule with the carbon atoms in the shell arranged in a network of pentagons and hexagons. In the experimentally observed and energetically most favorable "soccer ball" configuration, all twelve pentagons are isolated. Since C₆₀ is the smallest fullerene obeying the isolated pentagon rule, there were questions about the stability of smaller pure carbon cage

molecules formed from pentagons and hexagons since they must necessarily contain adjacent pentagons with a resultant large energy strain. Subsequently, a number of different size fullerenes have been synthesized in small amounts.

Under appropriate nonequilibrium growth conditions, carbon atoms form relatively stable hollow clusters of well-defined mass number, collectively known as fullerenes. The mass production of these clusters, and their purification and condensation into a solid are essential to full characterization and utilization of these new materials. While gas phase experiments indicate a wide range of fullerenes, beyond C₆₀ only a few pure fullerene solids have been obtained, notably C₇₀.

Lower mass fullerenes are of particular interest because their high curvature and increased energy strain owing to adjacent pentagon rings could lead to solids with unusual intermolecular bonding and electronic properties. These structures are expected to display chemical, electronic, magnetic and mechanical properties significantly different from, and possibly technologically more important than the more conventional C₆₀ fullerenes.

Summary of the Invention

Accordingly, it is an object of the invention to provide a solid state carbon material formed of fullerenes based on thirty six carbon atoms (C₃₆).

It is also an object of the invention to provide a method for making a solid state carbon material from C₃₆ fullerenes .

It is another object of the invention to provide an improved method for making fullerenes with enhanced C₃₆ yield.

It is a further object of the invention to provide other related materials based on C₃₆ fullerenes.

The invention is a solid phase or form of carbon based on fullerenes with thirty six carbon atoms (C₃₆). C₃₆ is a stable carbon cage molecule. The C₃₆ structure with D_{6h} symmetry is one of the two most energetically favorable, and is conducive to forming a periodic system. The lowest energy crystal is a highly bonded network of hexagonal planes of C₃₆ subunits with AB stacking. Electron diffraction indicates that C₃₆ forms a closed packed solid with a lattice constant significantly smaller than the C₆₀ counterpart. In contrast to solids formed from C₆₀ and higher order fullerenes, the C₃₆ solid is not a purely van der Waals solid, but has covalent-like bonding, leading to a solid with enhanced structural rigidity. The solid fullerene material may be termed a fullerite.

The invention includes the method of making the solid C₃₆ material by synthesizing and selecting out C₃₆ fullerenes in relatively large quantities. A C₃₆ rich fullerene soot is produced in a helium environment arc discharge chamber by operating at an optimum helium pressure (400 torr). The C₃₆ is separated from the soot by a two step process. The soot is first treated with a first solvent, e.g. toluene, to remove the higher order fullerenes but leave the C₃₆. The soot is then treated with a second solvent, e.g. pyridine, which is more polarizable than the first solvent used for the larger fullerenes. The second solvent extracts the C₃₆ from the soot. Thin films and powders can then be produced from the extracted C₃₆.

The invention also includes other materials based on C₃₆ fullerenes, providing for different properties. The smaller size of C₃₆ compared to C₆₀, and the higher percentage of 5-membered rings, make C₃₆ more reactive. Therefore other atoms can be attached to

the carbon atoms in the C₃₆ molecules. For example, the C₃₆ fullerene can be coated with F or Cl to make Teflon-like balls. Also, some of the carbon atoms can be substituted with other elements, e.g. nitrogen, which shortens some of the C-C bonds. In addition, the C₃₆ fullerene cages can trap a range of atoms. Endohedral binding energies indicate that C₃₆ may be the smallest fullerene which can easily trap a range of atoms. For the lowest energy solid, sodium appears to be the largest alkali atom that can be intercalated into the crystal structure without causing severe structural distortion.

Brief Description of the Drawings

In the accompanying drawings:

Fig. 1 is a mass spectrum of an arc discharge carbon soot film grown at 400 torr helium.

Fig. 2 shows an arc chamber apparatus for producing C₃₆ fullerenes.

Fig. 3 is a mass spectrum of the product resulting from the C₃₆ purification of a carbon soot material produced according to the invention.

Fig. 4 shows an extractor for extracting C₃₆ under inert conditions.

Figs. 5A, B are theoretical and observed NMR spectra of C₃₆.

Figs. 6A, B show the structures of the D_{6h} and D_{2d} C₃₆ molecules.

Figs. 7A, B respectively show a top view of the repeating planes and a side view showing the relaxed interlayer bonding for the AB stacking sequence.

Figs. 7C, D respectively show a top view of the repeating planes and a side view showing the relaxed interlayer bonding for the ABC stacking sequence.

Fig. 8A shows a top view of a planar bonded layer of C₃₆ units (S₂ sheet).

Figs. 8B-D show side views of relaxed crystal structures formed by stacking S2 layers according to AA, AB, or ABC sequences respectively.

Fig. 9a shows a $C_{24}N_{12}$ fullerene molecule.

Fig. 9B shows a D_{6h} $C_{36}Cl_{12}$ molecule.

Detailed Description of the Invention

A. Preparation of C_{36} Fullerenes

Bulk quantities of C_{36} fullerenes are produced by a modified Kratschmer-Huffman arc plasma technique. The technique has been modified to enhance the production of C_{36} . The graphite arc soot produced by a carbon arc discharge under normal conditions contains fullerenes of different mass, particularly C_{60} and other higher order fullerenes. Very little, if any, of the lower order fullerenes are produced. However, as a part of the invention, it has been determined that under certain conditions, C_{36} can be produced in relatively large amounts, e.g. 1-2% of the carbon soot.

Optimum parameters for C_{36} production were determined in a helium environment arc discharge chamber originally designed for C_{60} production. An arc was started between two 1/4 inch diameter graphite electrodes, using DC current of 100 A while maintaining a 1 mm gap between the electrodes. Arcing was maintained for several minutes until a uniform carbon film of about 10 μ m thickness of the graphite arc soot was deposited on a substrate placed 10 cm from the discharge region on the wall of the water cooled chamber.

The mass spectrum of an arc discharge carbon soot film grown in 400 torr helium is shown in Fig. 1. The dominant peaks are at 720 and 432 atomic mass units (amu), which

correspond to 60 carbon atoms and 36 carbon atoms respectively. The C₃₆ and C₆₀ peaks are of comparable magnitude and these molecular species appear to be the most prominent in the sample. Lesser peaks are also observed, e.g. at 840 amu (C₇₀).

The synthesis of C₃₆ is very sensitive to operational parameters, notably helium pressure. A series of experimental runs was carried out at different fixed static helium pressures between 50 and 1500 torr. Runs in the series at pressures significantly different from 400 torr failed to produce prominent peaks below 720 amu in the mass spectrum. To produce bulk amounts of C₃₆ suitable for purification, arcing runs in 400 torr helium were repeated and the resulting soot was collected from the chamber walls.

To prevent exposure of the C₃₆ to air prior to purification (which affects its solubility) an arc chamber 10, shown in Fig. 2, was designed which could be transferred into an argon glovebox prior to opening. Arc chamber 10 has an inner chamber 12 where the arc takes place and an outer chamber 14 which is filled with a water cooling bath. Inner chamber wall 16 is mounted to outer chamber wall 18, e.g. at top flange 20. Water is flowed, through inlets/outlets 22, 24, through chamber 14 defined between walls 16, 18.

Positive electrode (anode) 26 is mounted on and extends through cover 28 into chamber 12. To eliminate a similar other electrode, grounded chamber wall 16, e.g. made of stainless steel, is used to ground a graphite plate 30 at the bottom of chamber 12. Thus graphite plate 30 acts as the cathode and provides a surface to arc the typically 1/4 inch graphite anode 26 against. Anode 26 and chamber wall 16 are connected to a power supply 32 (e.g. DC or AC, 30-200 A, 0-70 V) to provide arc current, with wall 16 connected to the ground terminal 34.

Chamber 12 is connected through valve 36 to vacuum pump 38 to evacuate the system. Chamber 12 is also connected through valve 40 to a helium supply 42 which provides helium at the desired pressure. After the arc run, inner chamber 12 is removed from outer chamber 14, disconnected from pump 38 and helium supply 42, and transferred to an argon glovebox. Once inside the glovebox, inner chamber 12 is opened, and the soot is collected from wall 16.

B. Purification of C₃₆ Fullerenes

Once the carbon soot containing bulk quantities, e.g. 1-2%, of the C₃₆ fullerenes has been collected, the C₃₆ must be extracted or purified. A two step process is used. One particular process makes use of the different solubilities of the fullerenes.

While C₆₀ and the higher order fullerenes are soluble in toluene or benzene, C₃₆ is not. Therefore, the collected soot is initially washed in toluene in a standard Soxhlet extractor to remove the higher order fullerenes.

According to the invention, C₃₆ has been found to be soluble in both pyridine (C₅H₅N) and carbon disulfide (CS₂), two very polar organic solvents. Since the C₃₆ dissolves slowly in the solvents, because of its strong covalent bonding, heating and ultrasound can be used to assist in obtaining a saturated solution.

Therefore, the toluene insolvent residue left in the extractor after removal of the higher order fullerenes is further processed with pyridine as the extracting solvent. The pyridine soluble extract (C₃₆) forms a yellow-brown solution while the toluene soluble extract (C₆₀, C₇₀) forms a red-brown solution. After evaporating the pyridine from the pyridine soluble extract, bulk solid C₃₆ material (powder) was produced.

As an alternative to the wet chemistry dual solvent purification method, the second step, i.e. the removal of the C₃₆ from the remaining toluene insoluble material, can be done by thermal evaporation (sublimation purification). After the first step of toluene solvent extraction, the toluene insoluble material was dried in vacuum at 150°C for two hours and placed in the tungsten boat of a thermal evaporator. The evaporator boat was heated under a pressure of less than 1 μ torr, ultimately reaching a temperature of more than 1500°C. In about 20 minutes, films of about 100 nm thickness were deposited on a metal substrate suspended about 5 cm over the boat.

A mass spectrum of the material purified from the toluene insoluble extract of the carbon arc soot is shown in Fig. 3. The spectrum shows a single peak at 36 carbon atoms and no signal in the higher mass end of the spectrum, indicating that the material is composed of C₃₆ molecules.

An extractor 50 to extract fullerenes from graphite arc soot under inert conditions is shown in Fig. 3. Extractor 50 has a main tube 52 and a side tube 54 connected between the top and bottom of the main tube 52. The major modification in the extractor 50 is the inclusion of high vacuum Teflon stopcocks 56, 58 at the bottom and in the side tube. Water jacket 60 also surrounds main tube 52, reducing its length. A coarse frit 62 is placed across the main tube 52. The top of the extractor 50 is a female fitting 64 and the bottom is a male fitting 66.

C. Crystal Structure

The purified solid C₃₆ material obtained was characterized using C-13 NMR, bulk electron diffraction, mid-infrared transmission, and solid state transport studies. For a 36 atom carbon cage with hexagonal and pentagonal faces, 15 different isomeric structures

are theoretically possible. Calculations indicate that the lowest energy isomers are structures with D_{6h} and D_{2d} symmetry. These two molecules can be distinguished by NMR spectroscopy. Mass spectrometry using a laser desorption/time of flight mass spectrometer shows that the material is C_{36} , as shown in Fig. 2.

Fig. 5A shows the predicted molecular NMR spectra for the isolated D_{6h} and D_{2d} isomers (along with schematic structure drawings). Fig. 5B shows the observed NMR spectrum for C_{36} powder, obtained using a Chemagnetics 500 MHz instrument with magic-angle spinning. Taking into account broadening and shielding effects, the experimental spectrum suggests D_{6h} symmetry for the C_{36} molecule.

The 36 atom carbon clusters lie in an interesting size regime, intermediate between the closed cage fullerene ($N = 40-90$) and ring ($N = 10-28$) dominated sizes. Six distinct equilibrium structures have been considered: the D_{6h} , D_{2d} , C_{2v} , and D_{3h} fullerenes; a C_{3v} corannulene-like bowl; and a D_{18d} monocyclic ring. Eleven other fullerenes structures are theoretically possible but very unlikely to form due to excessive pentagon clustering. The D_{6h} and D_{2d} fullerenes are predicted to be the most energetically favorable and stable.

The D_{6h} C_{36} structure, shown in Fig. 6A, is the most preferred since it has the higher symmetry and can form simple solids. In the D_{6h} structure, the pentagons form two belts around the top and bottom hexagons, and there is a row of hexagons separating these pentagon belts. The relaxed D_{6h} C_{36} structure has a height of 5.2 Å and a width of 4.9 Å as measured from the atomic positions. The symmetry unique or inequivalent atoms C1, C2, C3 are labelled in Fig. 6A.

The D_{2d} C_{36} structure, shown in Fig. 6B, is also preferred.

The solid state materials formed of the C₃₆ fullerenes may have a hexagonal or rhombohedral crystal structure with the hexagonal structure being most preferred. The crystal with hexagonal symmetry is substantially lower in energy, and has different electronic properties. Electron diffraction was used to investigate the crystal structure of solid C₃₆. A small amount of solid C₃₆ was ground up, dispersed on a holey carbon grid, and inserted into a JEOL 200X transmission electron microscope (TEM). The material was observed to be polycrystalline with a grain size of about 100 nm. The TEM diffraction pattern was hexagonal, suggesting a close packing arrangement perpendicular to the zone axis. The calculated d-spacing was 6.68 Å.

The hexagonal and rhombohedral crystal symmetries is shown in Figs. 7A-D. Each is formed by stacking hexagonally symmetric planes of well separated C₃₆ units. Figs. 7A-D show two kinds of stacking sequences, AB and ABC, corresponding to the hexagonal and rhombohedral crystals respectively. Figs. 7A, B respectively show a top view of the repeating planes and a side view showing the relaxed interlayer bonding for the AB stacking sequence. Figs. 7C, D respectively show a top view of the repeating planes and a side view showing the relaxed interlayer bonding for the ABC stacking sequence. The fundamental stacking unit is a plane of unbonded C₃₆ molecules referred to as sheet 1 (S1) and the two crystals are labelled S1-AB and S1-BC for the two kinds of stacking. The C₃₆ units are essentially noninteracting within an S1 sheet. (The AA stacking sequence (S1-AA) is not considered because it does not form a metastable structure.)

The D_{6h} C₃₆ molecules form stable dimers and trimers with two or six intermolecular bonds respectively. Further C₃₆ molecules may be added in the same

manner to form a sheet of bonded units referred to as sheet 2 (S2). Different crystal structures may also be formed by stacking S2 layers. Fig. 8A shows a top view of a planar bonded layer of C₃₆ units (S2 sheet), and Figs. 8B-D show side views of relaxed crystal structures formed by stacking S2 layers according to AA, AB, or ABC sequences respectively.

For S2-AA there is bonding between all six carbon atoms of the hexagon rings on top and bottom, as in the case of S1-AB. Stacking S2 sheets in the AB sequence results in a slightly larger density and a substantially larger binding energy than S2-AA. It is the lowest energy crystal structure. The S2-ABC contains no interlayer bonding and is not likely to be stable.

The C₃₆ solid is not a purely van der Waals solid, but has covalent bonding which produces a solid with enhanced structural rigidity.

D. Properties and Related Compounds

Because of its higher curvature and greater percentage of pentagons, the C₃₆ is more reactive than C₆₀. Thus it can produce compounds. Because of the covalent bonding of the solid material, very hard polymerized solids are formed. Also because of the high curvature, the electron-phonon coupling is enhanced, leading to high superconducting transition temperatures.

The related materials based on C₃₆ fullerenes include (1) substitutional doping of carbon atoms on the fullerene cages by electron donors and acceptors such as N or B; (2) addition of other atoms, e.g. halogens, to the carbon atoms; and (3) endohedral doping of the fullerene molecules. All of these may significantly change the properties of the material.

Several N substitutionally doped C₃₆ D_{6h} cages have been considered: C₃₄N₂, C₂₈N₈, and C₂₄N₁₂. In all cases no two dopant atoms are placed next to one another since the strong nitrogen dimer bond would weaken the overall structure. The most symmetric configurations are the most energetically favorable. The highest symmetry case is C₂₄N₁₂, shown in Fig. 9A, which retains the full D_{6h} symmetry. The substitution of N atoms for C atoms in the cage changes the properties of the material, e.g. the C-C bonds across the hexagon belt are shortened by about 10%.

Thus the invention encompasses C₃₆ fullerene materials where the material has the more general formula C_{36-a}X_a, where X is the doping atom, e.g. N or B, and $0 \leq a \leq 12$.

Other atoms can also be added to the C₃₆ fullerene by binding to the carbon atoms in the cage. For example, the C₃₆ fullerenes may be halogenated, eg. fluorinated or chlorinated. The D_{6h} C₃₆Cl₁₂ molecule is shown in Fig. 9B.

The invention also includes endohedrally doped fullerenes and intercalated solids. An atom M may be placed in the center of a C₃₆ molecule (M @ C₃₆). Binding energy calculations show that endohedral doping of C₃₆ with alkali earth atoms Mg, Ca, and Sr, and also with Si, Ge, and Zr, is possible. The C₃₆ fullerene appears to be the smallest size carbon cage which can easily trap additional atoms. As far as intercalation into the solid material, the S₂-AB crystal structure has two cavities per C₃₆ which can be doped with other atoms, e.g. Na or K. However, because of the ionic radii, Na appears to fit well but K causes structural changes.

Changes and modifications in the specifically described embodiments can be carried out without departing from the scope of the invention which is intended to be limited only by the scope of the appended claims.

Claims

1. A composition of matter comprising a solid state material formed of C₃₆ fullerene molecules.
2. The composition of Claim 1 wherein the solid state material has a crystalline structure.
3. The composition of Claim 2 wherein the crystalline structure is hexagonal.
4. The composition of Claim 1 wherein the molecules are covalently bonded.
5. An article of manufacture comprising a thin hard coating formed of a solid state C₃₆ material.
6. A method of making a solid state C₃₆ fullerene material, comprising:
producing C₃₆ rich graphite arc soot;
removing higher order fullerenes from the soot, leaving a C₃₆ containing residue;
removing the C₃₆ from the residue;
producing a solid material from the C₃₆ removed from the residue.

7. The method of Claim 6 wherein the C₃₆ rich soot is produced by operating a carbon arc discharge in a helium atmosphere at about 400 torr pressure.

8. The method of Claim 6 wherein the higher order fullerenes are removed using a first solvent and the C₃₆ is removed using a second solvent.

9. The method of Claim 8 wherein the first solvent is toluene and the second solvent is pyridine.

Abstract of the Disclosure

A solid phase or form of carbon is based on fullerenes with thirty six carbon atoms (C₃₆). The C₃₆ structure with D_{6h} symmetry is one of the two most energetically favorable, and is conducive to forming a periodic system. The lowest energy crystal is a highly bonded network of hexagonal planes of C₃₆ subunits with AB stacking. The C₃₆ solid is not a purely van der Waals solid, but has covalent-like bonding, leading to a solid with enhanced structural rigidity. The solid C₃₆ material is made by synthesizing and selecting out C₃₆ fullerenes in relatively large quantities. A C₃₆ rich fullerene soot is produced in a helium environment arc discharge chamber by operating at an optimum helium pressure (400 torr). The C₃₆ is separated from the soot by a two step process. The soot is first treated with a first solvent, e.g. toluene, to remove the higher order fullerenes but leave the C₃₆. The soot is then treated with a second solvent, e.g. pyridine, which is more polarizable than the first solvent used for the larger fullerenes. The second solvent extracts the C₃₆ from the soot. Thin films and powders can then be produced from the extracted C₃₆. Other materials are based on C₃₆ fullerenes, providing for different properties.

Total counts (arbitrary linear units)

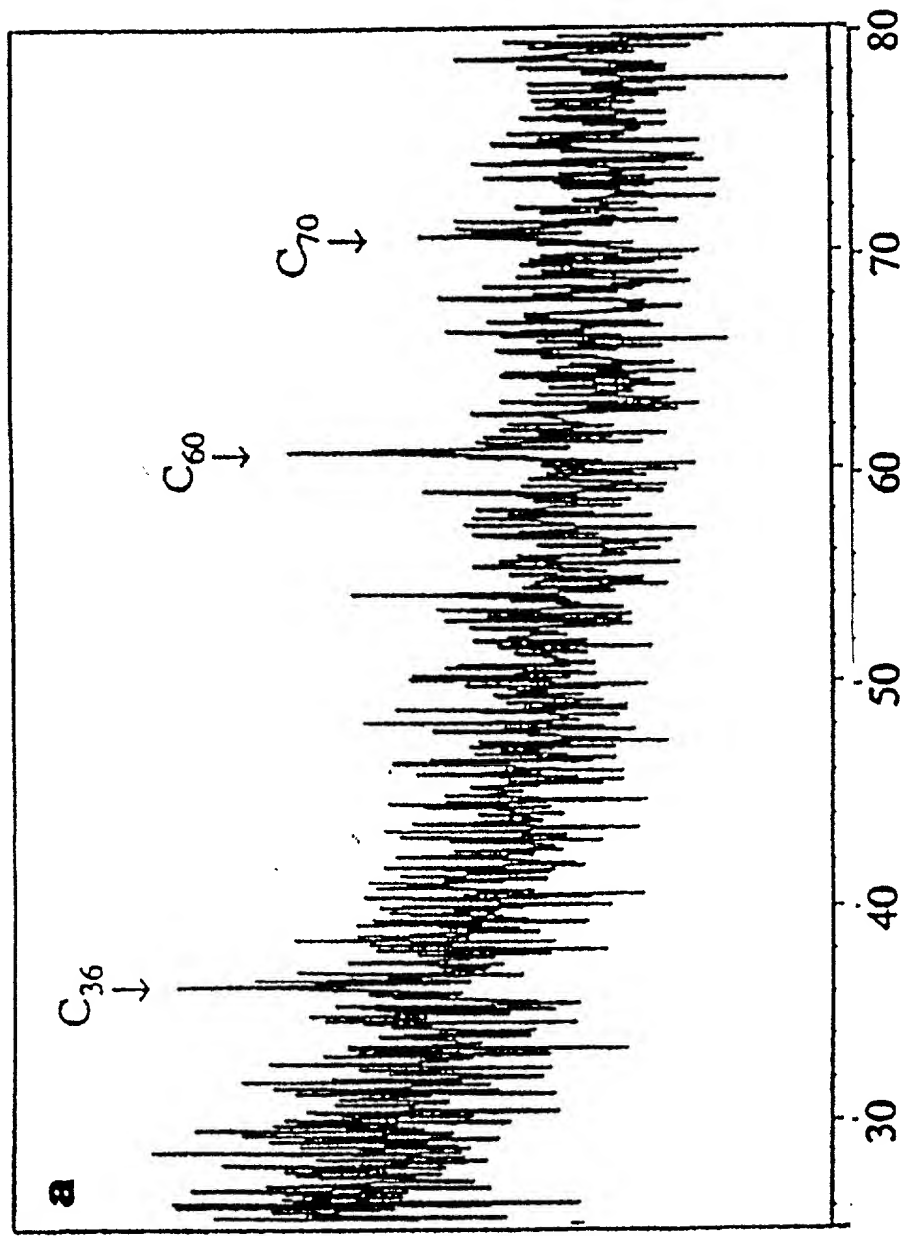
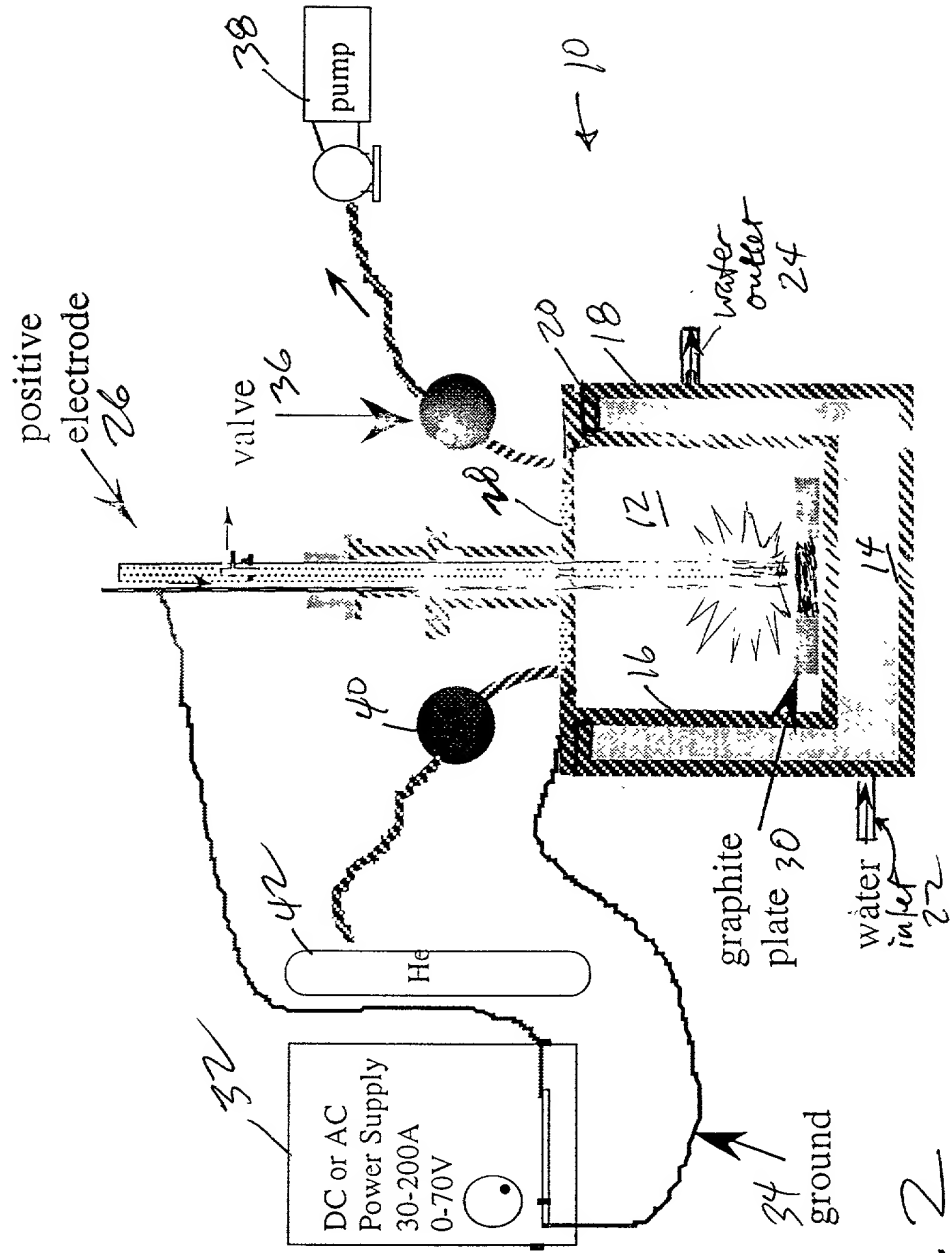
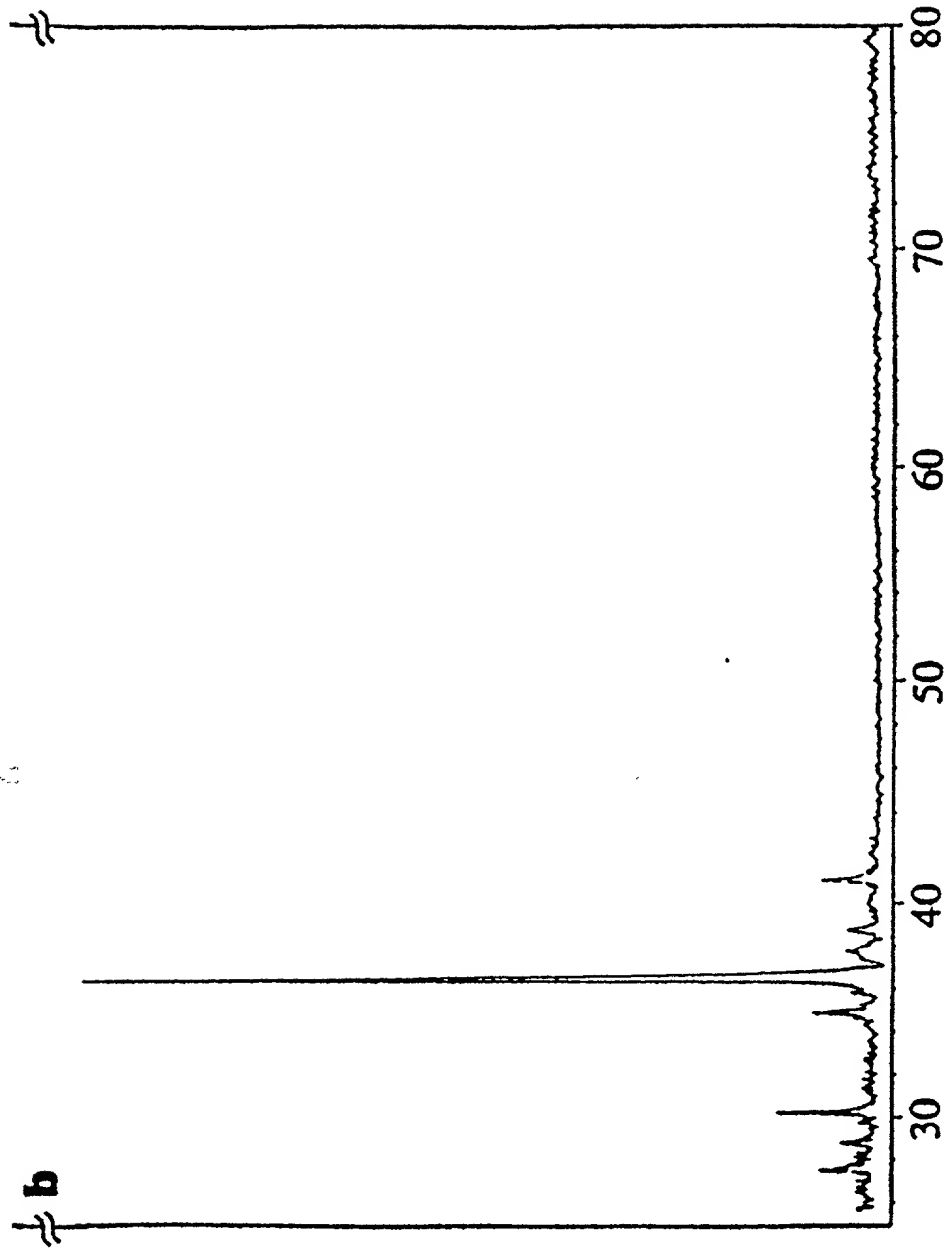


Fig. 1



[illegible]

Number of carbon atoms

Fig. 3

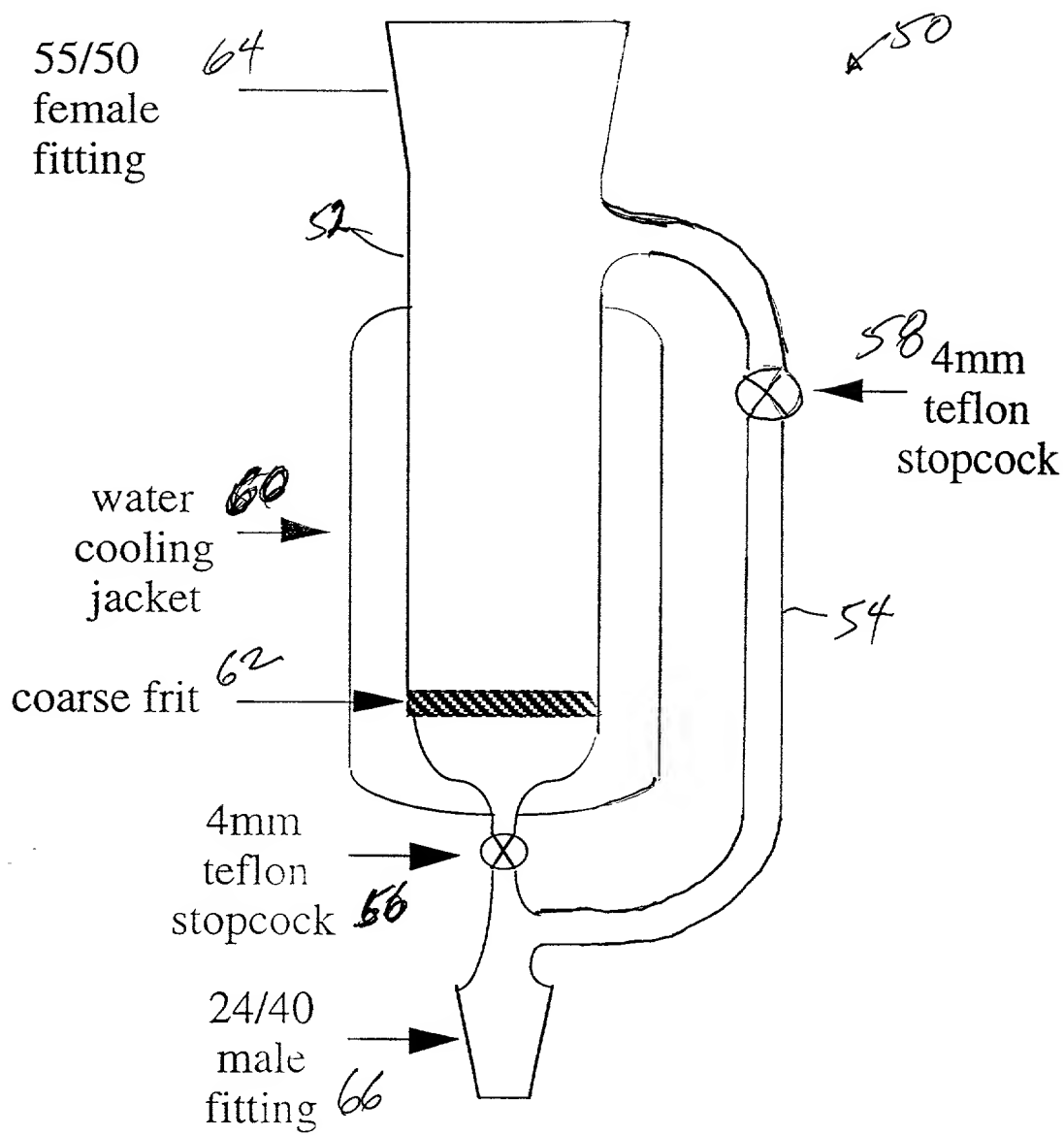


FIG. 4

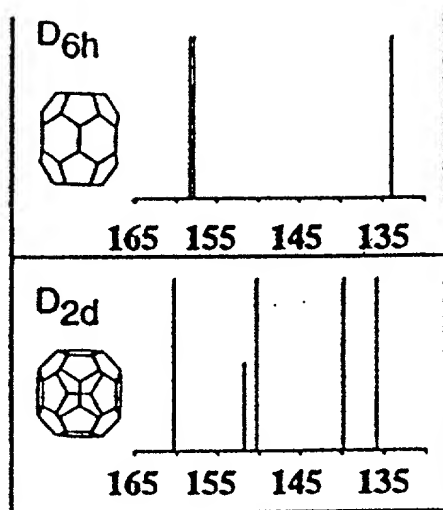


FIG. 5A

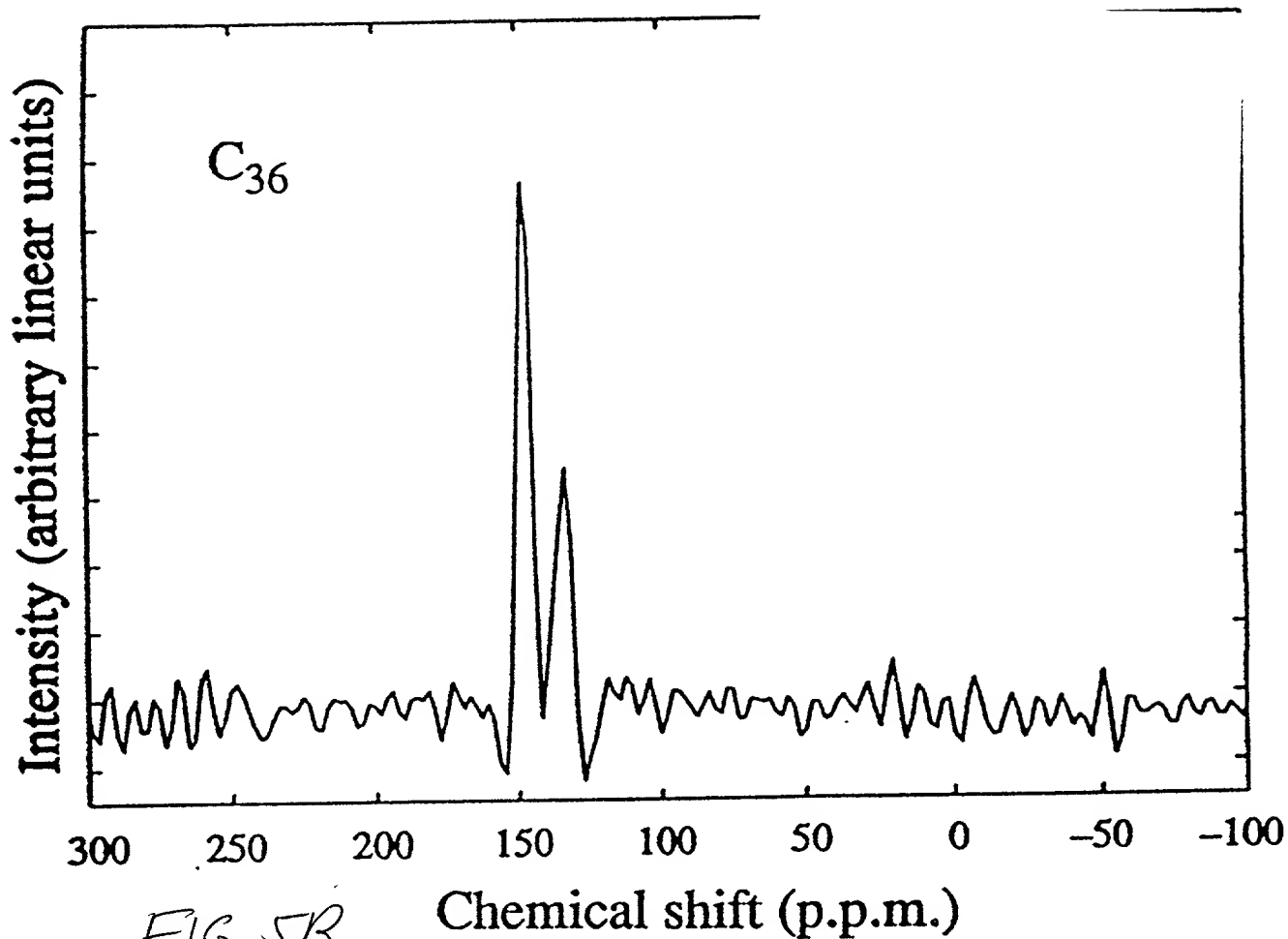


FIG. 5B

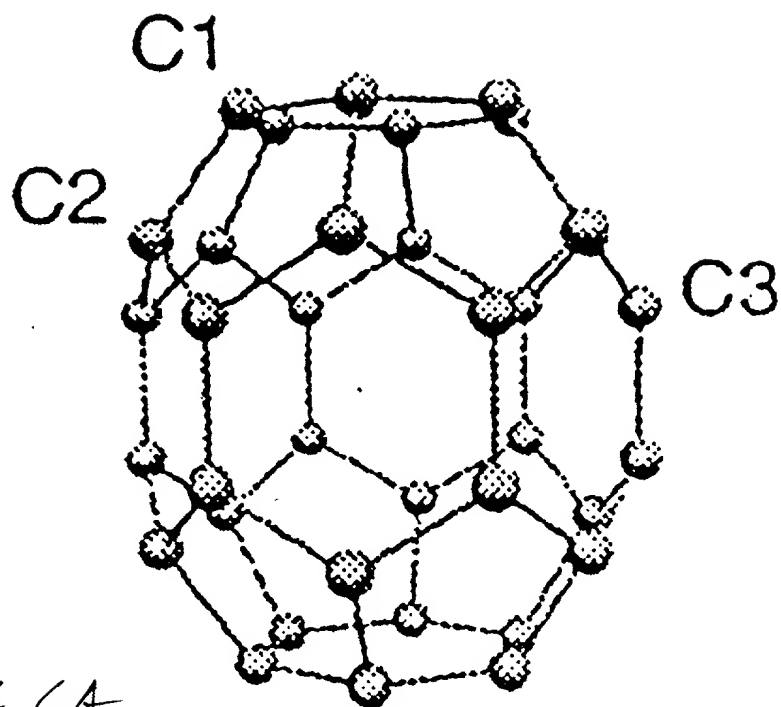


FIG. 6A

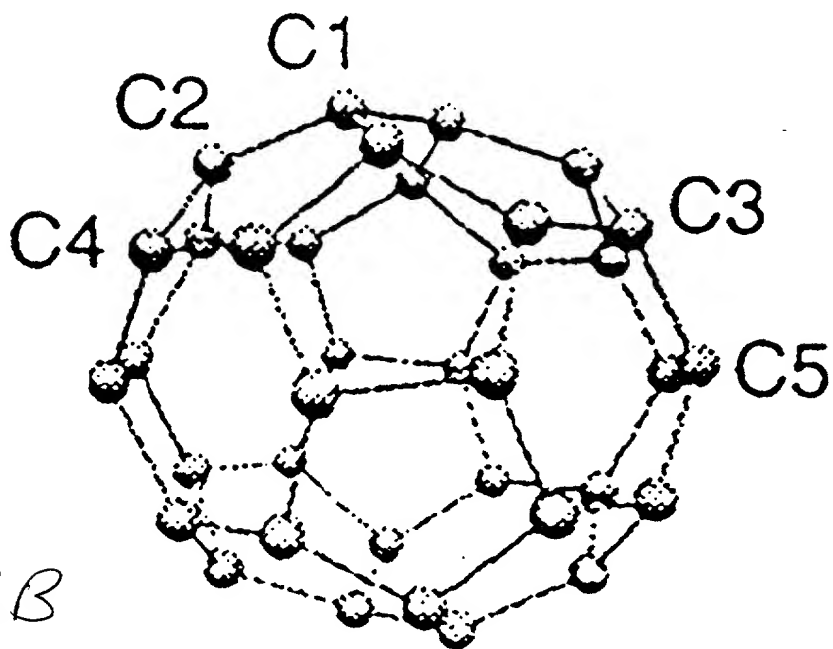
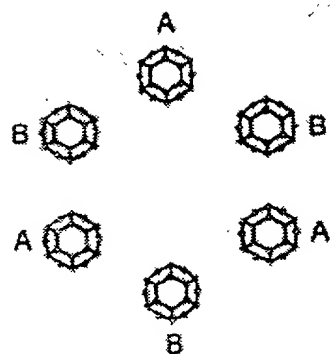
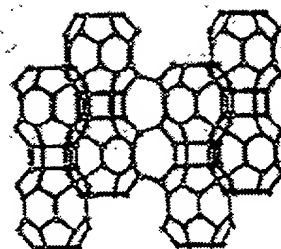


FIG. 6B



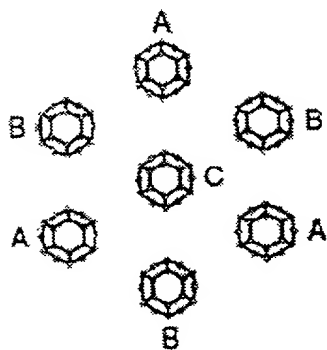
2XS1

FIG. 7A



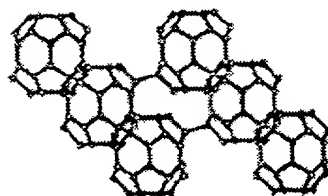
S1-AB

FIG. 7B



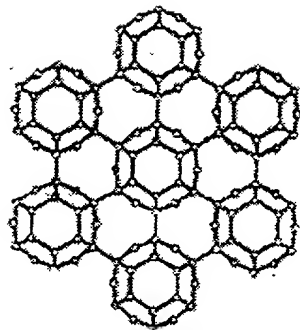
3XS1

FIG. 7C



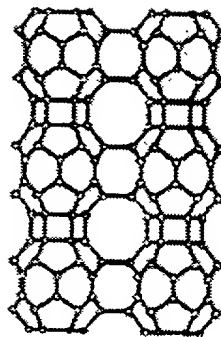
S1-ABC

FIG. 7D



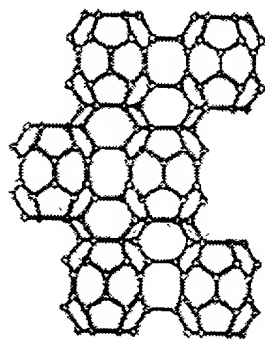
S2

FIG. 8A



S2-AA

FIG. 8B



S2-AB

FIG. 8C



S2-ABC

FIG. 8D

00548089 030300

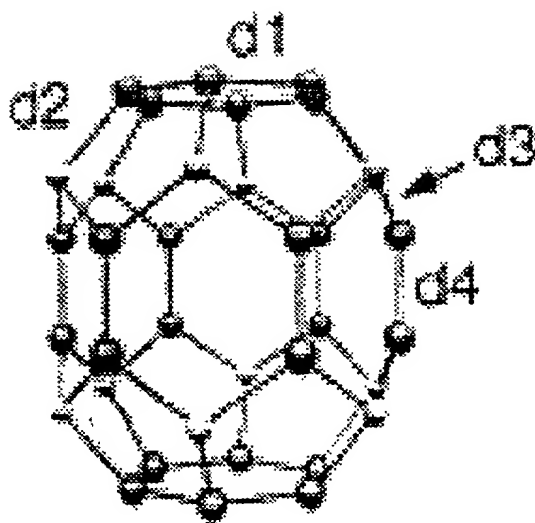


FIG. 9A

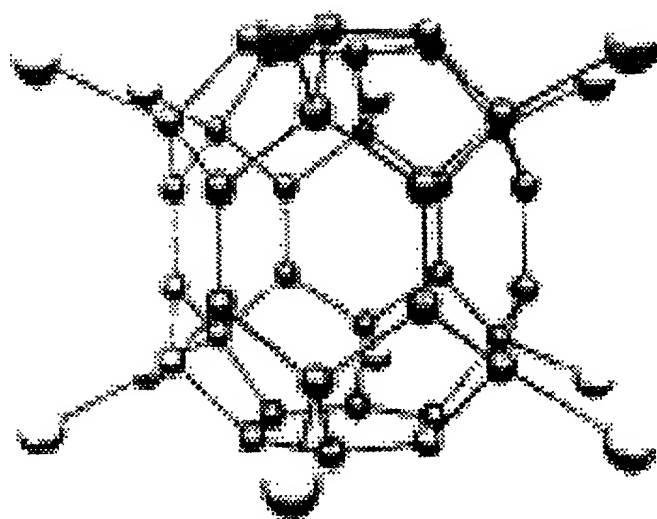


FIG. 9B